ECENED TO TO

THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF:

Shigeki NAKATSUKASA, Hiroyuki OHGI and Kazuyori YOSHIMI

: GROUP ART UNIT: 1752

SERIAL NO: 08/974,490

: EXAMINER: Barbara Gilmore

FILED: November 19, 1997

FOR: Resin composition and multilayered structure

DECLARATION UNDER 37 C.F.R. § 1.132

Honorable Commissioner of Patents and Trademrks,

Washington, D.C. 20231

Sir:

I, Kazuyori Yoshimi, residing at 1621, Sakazu, Kurashiki-city, Okayama Prefecture, Japan do hereby declare as follows:

I have graduated Okayama University, Department of Science, Master Course in March, 1970; and from April, 1970 up to date I have been an employee of KURARAY CO., LTD. and have been mainly engaged in the field of development of ethylene-vinyl alcohol copolymer (hereinafter referred to as EVOH). I am familiar with the instant technical field. And I am a third inventor of the present application.

I declare that the results of experiment I have conducted are as follows and that they are true to the best of my knowledge and belief.

In order to show flexural fatigue resistance and impact resistance of the resin composition comprising EVOH and high density polyethylene (HDPE), experiment has been carried out under my supervision as follows.

Experiment 1

A dry blend was prepared from the following two components.

(A) High density polyethylene, "HI-ZEX 8200B" from Mitsui Chemicals, Inc.

(10 parts by weight)

Density = 0.956 g/cm^3

MFR = 0.03 g/10 min (190°C, 2,160 g load)

Ultimate Tensile (ASTM D-638) = 40 MPa

(B) EVOH (90 parts by weight)

Ethylene content = 32 mol%

Degree of hydrolysis = 99.6%

MFR = $3.1 \text{ g/}10 \text{ min } (210^{\circ}\text{C}, 2,160 \text{ g load})$

Content of phosphorus compound (potassium dihydrogenphosphate) = 100 ppm (in terms of phosphorus element)

Content of potassium = 125 ppm (in terms of potassium element)

Content of sodium salt (sodium acetate) = 65 ppm (in terms of sodium element)

The dry blend was pelletized by extrusion through an extruder (40 mm in diameter, L/D = 24, compression ratio = 3.8) having a Madock-type mixing zone. Thus there was obtained the resin composition.

A 5-layered film specified below was produced by co-extrusion from the resin composition and the following two components fed into separate extruders.

Layer construction:

LLDPE/AD/RC/AD/LLDPE = $50/5/20/5/50 \mu m$

Total thickness = $130 \mu m$

• LLDPE: Ethylene-α-olefin copolymer produced by using a conventional Ziegler catalyst ("UF420" from Mitsubishi Chemical Corporation)

density = 0.925 g/cm^8

MFR = $0.8 \text{ g/}10 \text{ min } (210^{\circ}\text{C}, 2,160 \text{ g load})$

• AD: Linear low-density polyethylene graft-modified with maleic anhydride for the adhesive resin layer ("Admer NF500" from Mitsui Chemicals, Inc.)

MFR = $3.6 \text{ g/}10 \text{ min } (210^{\circ}\text{C}, 2,160 \text{ g load})$

• RC: Resin composition

Each component was extruded under the following conditions.

- LLDPE: at 200 to 240°C through a 65-mm single-screw extruder with an L/D ratio of 22.
- AD: at 160 to 220°C through a 40-mm single-screw extruder with an L/D ratio of 26.
- Resin composition: at 200 to 240°C through a 40-mm single-screw extruder with an L/D ratio of 26.

The melt was discharged from a feed block die (600 mm wide) at 240°C.

A specimen (12 x 8 inches) was cut out of the resulting multilayered film and conditioned at 20°C and 65 %RH. The specimen was made into a cylinder, 3.5 inches in diameter. The cylinder was held by grips at its ends on a Gelbo Flex Tester (made by Rigaku Kogyo). Initially, the grips were 7 inches apart. The specimen was twisted by turning the grips in opposite direction through an angle of 440° such that the distance between the grips was decreased to 3.5 inches. Then the grips were moved straight and horizontally so that the distance between them

was decreased to 2.5 inches. Finally, the grips were returned to their original positions. (When the specimen was flexed to the utmost limit, the grips were 1 inch apart.) These steps were repeated at a rate of 40 cycles per minute. This test was carried out at 20°C and 65 %RH. Pinholes appeared in the specimen when the specimen was observed after 500 cycles.

A sample of flexible container was prepared by heat sealing from the multilayered film. (Two sheets of film measuring 20 by 30 cm were placed one over the other and three sides were heat-sealed, with the 20-cm side left open.) This sample was filled with water (20°C) and the open side was heat-sealed. In this way there was obtained a flexible container for bag-in-box. This container was dropped on a concrete floor. The height for the container to break (allowing water to leak) was recorded. This test was repeated for 30 samples and the results were calculated according to JIS K7211 (Section 8 for calculations) to obtain the height for 50% of the samples to break. The height for the container to break was 1.1 m.

I, the undersigned declarant, declare further that all statement made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and; further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001, of Title 18, of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed this Zb th day of December, 2001

Name:

Kazuyori Yoshimi

Kazuyori Yoshimi



ENGAGE

EG 8100

Polyolefin Elastomer for General Purpose Elastomeric Applications

• General purpose elastomer

- Excellent impact properties in blends with polypropylene and polyethylene
- Excellent flow characteristics
- Excellent heat aging and compression set when crosslinked

ENGAGE* EG 8100 polyoletin elastomer is a saturated ethylene-octene copolymer that provides excellent flow properties and is efficiently crosslinked by peroxide, silane or irradiation. ENGAGE EG 8100 POE delivers exceptional heat aging, compression set, and weather resistance properties. This elastomer is also highly effective as an impact modifier in polyoletins.

| Physical Properties | Test Method | VALUES ¹ |
|--|------------------|---|
| Density, gm/cc | ASTM D-792 | .87 |
| Mooney Viscosity, ML 1+4 @ 121°C | ASTM D-1646 | . 23 |
| Percent Comonomer, octene | Dow ⁸ | 24 |
| Melt Index, dg/min | ASTM D-1238 | 1 |
| Melt Flow Ratio, 1,0/I2 | ASTM D-1238 | 7.5 |
| Dow Rheology Index (DRI) | Dows | 2 |
| Ultimate Tensile, psi (MPa) | ASTM D-638 | 1,500 (10.3) |
| 100% Tensile Modulus, psi (MPa) | ASTM D-638 | 350 (2.4) |
| Ultimate Elongation, % | ASTM D-638- | 800 |
| Hardness, Shore A | ASTM D-2240 | 75 |
| Brittleness Temperature, °C | ASTM D-746 . | <-76 |
| Flexural Modulus, 2% Secant, psi (MPa) | ASTM D-790 | 2,340 (16.1) |
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^{*}Trademark of The Dow Chemical Company.

- See "Handling Considerations" on the reverse side.



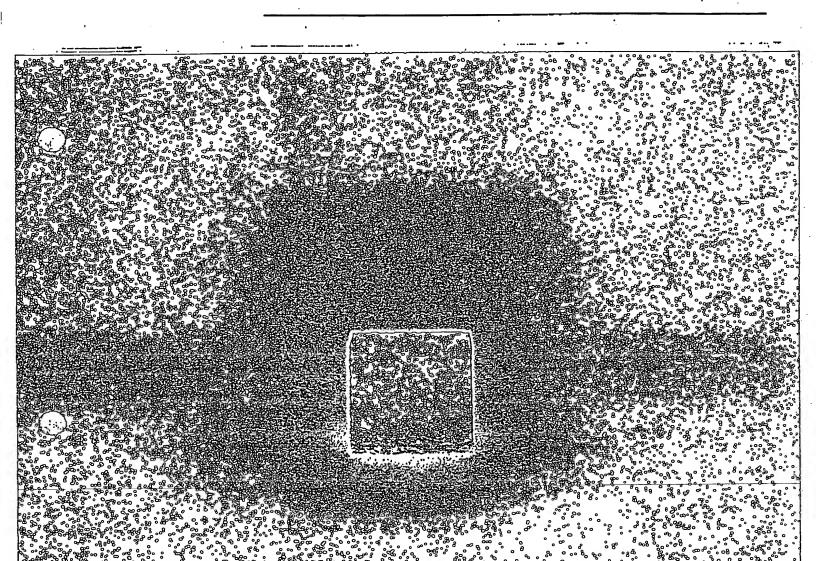
There are typical properties only, and are not to be regarded as sales specifications.
Thated on ASTM (\$2236, Method 0.

³A calculated value hased on complex viscosity that expresses the relative influence of long chain branching on the extratability of homogeneous (single alteratalyst) polyclefas.

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| | characteristic | Temperature | melting point | temperature | Vicat softening | resistance | stress crack | Hardness | Strength | Izod impact | Olsen hardness | Elongation | Ultimate | Ultimate Tensile | Stress at yield | density | Melt flow rate | Properties | |
| main use | | C | ್ ದ | | ದೆ | | hour | | | J/m | MPa | | % | MPa | MPa | kg/m³ | g / 10 min. | unit | |
| | | DJapa | D2117 | | D1525 | | D1693 | D2240 | | D256 | D747 | | D638 | D638 | D638 | D1505 | D1238 | test method (ASTM) | |
| | | | | | ı | | 1 | Shore D | | 1 | - | and the second | I | 1 | 1 | 1 | 190°C | test conditions | |

VERIFICATION OF TRANSLATION

I, Takahiro Sekiguchi of c/o Kuraray Co., Ltd. 1621, Sakazu, Kurashiki-City, Okayama-Pref., Japan

declare as follows:

- 1. That I am well acquainted with both the English and Japanese languages, and
- 2. That the attached document is a true and correct translation made by me to the best of my knowledge and belief of:

a part of catalog of HI-ZEX (Mitsui Chemical Corporation).

December 27, 2001

(Date)

Takahiro Lekiquehi
(Signature of Translator)